Chemistry of Natural Compounds, Vol. 31, No. 2, 1995

ALKALOIDS OF *Aconitum nemorum*

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UDC 547.944/945

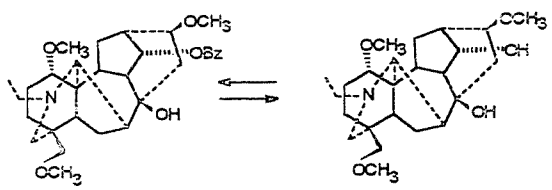
Talatisamine, 14-acetyltalatisamine, and the new base 14-benzoyltalatisamine have been isolated from the roots of Aconitorum nemorum. *A structure of benzoyltalatisamine has been proposed on the basis of a study of spectral characteristics and chemical transformations and has been confirmed by partial synthesis.*

We have investigated the alkaloids of the roots of *Aconitum nemorum* M. Pop., gathered in the period of seed-ripening in the environs of the village of Santash (Kyrgyzstan). The usual chloroform extraction followed by separation into ether and chloroform fractions yielded 0.47% of total alkaloids.

Talatisamine, 14-acetyltalisamine, and a base called nemorine and characterized by its empirical formula have been isolated from this plant previously [1, 2].

Continuing a study of the alkaloids of this plant, in addition to talatisamine and 14-acetyltalatisamine we have isolated a new base with the composition of $C_{31}H_{43}NO_6$ (**1**). The IR spectrum of the alkaloid had the absorption bands of hydroxy and ester groups and of ether bonds. According to its PMR spectrum, the alkaloid contained an N-ethyl and three methoxy groups and five aromatic protons in a monosubstituted benzene ring. The mass spectrum showed that it belonged to the lycoctonine group of alkaloids with a methoxy group at C-1 (maximum peak of the $M^+ - 31$ ion).

Alkaline hydrolysis of the alkaloid gave an amino alcohol identical with talatisamine (**2**). In the PMR spectrum of (**1**) the signal of C-14β proton was observed at 5.03 ppm (t, J = 5 Hz) and was displaced downfield by 1.00 ppm in comparison with the spectrum of talatisamine (**2**). This showed that the benzoyl group was located at C-14. The benzoylation of talatisamine (**2**) with benzoyl chloride in pyridine gave 14-benzoyltalatisamine, identical with the alkaloid isolated, which confirmed the conclusion drawn [3].



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EXPERIMENTAL

PMR spectra were taken on a Tesla BS-567 A 100 MHz instrument with HMDS as internal standard, mass spectra on a MKh-1310 instrument with a system for the direct injection of the sample into the ion source, and IR spectra on a UR-20 spectrometer in tablets with KBr.

For chromatography we used type KSK silica gel and deactivated alumina.

Isolation of the Total Alkaloids. Air-dried comminuted roots of the plant *Aconitum nemorum* (540 g) were wetted with a 5% solution of Na_2CO_3 and extracted with chloroform in a Soxhlet apparatus. The resulting chloroform extract (1.2

Institute of the Chemistry of Plant Substances, Academy of Sciences of the Republic of Uzbekistan, Tashkent, fax (3712) 89 14 75. Translated from Khimiya Prirodnykh Soedinenii, No. 2, pp. 283-284, March-April, 1995. Original article submitted November 21, 1994.

0009-3130/95/3102-0233$12.50

liter) was shaken with 5% H_2SO_4 (5 × 150 ml). The combined acid extracts were made alkaline by the addition of soda, with cooling, and were extracted first with ether and then with chloroform. After the solvents had been distilled off, 2.46 g of ether-extracted and 0.12 g of chloroform-extracted alkaloids were obtained.

Separation of the Ether-Extracted Alkaloids. The ether-extracted alkaloids (2.46 g) were chromatographed on a column of alumina with elution by chloroform to which methanol was gradually added (100:1). Fractions 5-12 yielded 1.09 g of talatisamine. The chloroform fractions were rechromatographed on a column of alumina and elution by ether gave 0.14 g of 14-acetyltalatisamine and 0.09 g of 14-benzoyltalatisamine.

14-Benzoyltalatisamine (1). $C_{31}H_{43}NO_6$, melting point of the perchlorate 219-221°C (EtOH). IR spectrum (ν_{max}, cm^{-1}): 1100, 1590, 1720. PMR spectrum (δ, ppm): 1.03 (3H, t, J = 7 Hz, $N-CH_2-CH_3$), 3.09, 3.21, 3.21 (each 3H, s, 3 × OCH_3), 5.03 (1H, t, J = 5 Hz, H−14β), 7.56 and 7.88 (5H, m, Ar−H). Mass spectrum, *m/z* (%): M^+ 525 (19), 494 (100), 146 (23), 105 (27).

Alkaline Hydrolysis of 14-Benzoyltalatisamine. A solution of 0.05 g of (1) in 3 ml of a 5% solution of KOH in methanol was boiled for an hour. After the methanol had been eliminated, the residue was dissolved in 2% H_2SO_4 and the acid solution was washed four times with ether. Then it was made alkaline with soda and extracted with chloroform. The extracts were dried over sodium sulfate and distilled. The acid fraction yielded 0.005 g of benzoic acid, and the alkaline fraction 0.027 g of talatisamine.

Benzoylation of Talatisamine. A solution of 0.5 g of talatisamine in 6 ml of pyridine was treated with 0.5 ml of benzoyl chloride, and the mixture was kept at room temperature for 12 h. The pyridine and the excess of benzoyl chloride were distilled off in a rotary evaporator. The residue was dissolved in 2% of H_2SO_4 and the acid solution was washed three times with ether and was then made alkaline with soda and extracted with chloroform. The chloroform extract was dried over sodium sulfate and evaporated to dryness. The product was dissolved in ethanol, and an alcoholic solution of perchloric acid was added to give a weakly acid medium. The 14-benzoyltalatisamine perchlorate that deposited after some time was separated off (0.42 g), mp 219-221°C.

REFERENCES

1. N. K. Abubakirov and Z. I. Chupova, Zh. Obshch. Khim., **29**, 2454 (1959).
2. T. F. Platonova, A. D. Kuzovkov, and P. S. Massagetov, Zh. Obshch. Khim., **28**, 3126 (1958).
3. M. N. Sultankhodzhaev, Advances in the Investigation of Alkaloid-Bearing Plants [in Russian], Fan, Tashkent (1993).